## Antal Kerpely Doctoral School of Materials Science and Technology



# Performance of granulated foam glass in cement matrix composite material

**Thesis Booklet** 

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#### **Abstract**

The growing demand for sustainable construction materials has led to the exploration of lightweight aggregates as alternatives to traditional natural aggregates. Foam glass, a porous material made from recycled glass, offers a promising solution due to its low density, high thermal insulation, and environmental benefits.

This research investigates the feasibility of using granulated foam glass for the production of structural lightweight concrete. The first part of the study focuses on the foam glass itself, examining the effects of sintering parameters on its properties, the impact of adding water to the preform mixture to enhance initial particle bonding, and the novel use of cement factory soot as a foaming agent for the first time in foam glass production. Additionally, a new laboratory-scale method for fabricating granulated foam glass is proposed.

The second part of the study is dedicated to the application of foam glass in structural lightweight concrete. A mix design for producing structural lightweight concrete using foam glass is presented. Both granulated and crushed forms of foam glass were employed as aggregates, and the differences in their influence on concrete properties were analyzed. Two types of raw glass powders, virgin and waste glass, were used to manufacture the foam glass aggregates, and the effect of the glass source on concrete performance was also evaluated. Furthermore, a novel thermogravimetric analysis (TGA)-based method was developed to assess the reactivity of foam glass aggregates in alkaline environments.

The results demonstrated that cement factory soot can be effectively used as a waste-derived foaming agent for foam glass production. Additionally, it was shown that in heating microscopy analysis, tracking the change in projected area is more reliable than height changes for determining the maximum expansion temperature. The inclusion of water in the foam glass preform mixture improves the initial adhesion between raw particles without significantly affecting the final properties of the foam glass. The application of the Taguchi design of experiments method enabled efficient optimization with a reduced number of tests, yielding results comparable to those from full factorial designs.

Regarding the use of foam glass in concrete, the findings indicate that replacing 20 vol% of natural aggregates with foam glass aggregates can produce structural lightweight concrete with a compressive strength exceeding 17 MPa and a density below 2000 kg/m³. Finally, the TGA-based method was confirmed as a viable new technique for evaluating alkali-silica reaction (ASR) potential in concrete materials.

#### 1. Introduction

Concrete is the most widely used construction material globally, with an annual production of approximately 30 billion tons. Despite its widespread use, traditional concrete has several limitations, including high density, poor thermal insulation, and environmental concerns related to the extraction of natural aggregates. Lightweight concrete, which incorporates lightweight aggregates, offers a promising solution by reducing structural weight, improving thermal insulation, and enhancing sustainability.

Foam glass, a porous material made from recycled glass, has emerged as a viable lightweight aggregate. Its unique properties, such as low density, high thermal insulation, and fire resistance, make it an attractive alternative to traditional aggregates. However, the use of foam glass in structural concrete requires a thorough understanding of its production, properties, and performance in concrete applications.

This study investigates the production, properties, and application of foam glass granules in structural lightweight concrete. The research addresses key challenges, including optimizing foam glass production, evaluating its performance in concrete, and assessing its durability against alkali-silica reaction (ASR). The findings provide valuable insights into the feasibility of using foam glass as a sustainable and high-performance lightweight aggregate in construction.

#### 2. Literature review

Lightweight concrete has emerged as a sustainable alternative to conventional concrete, offering advantages such as reduced weight, improved thermal insulation, and enhanced fire resistance. Its density ranges from 400 to 2000 kg/m³, significantly lower than the 2400 kg/m³ of conventional concrete [1]. Lightweight concrete is categorized into three types: foamed concrete, no-fines concrete, and lightweight aggregate concrete. Among these, lightweight aggregate concrete is the most versatile, with densities ranging from 500 to 2000 kg/m³ [2]. The use of lightweight aggregates, such as expanded clay or slate, reduces the overall weight while maintaining structural integrity [2]. However, a significant knowledge gap exists in the development of structural lightweight concrete using granulated and crushed foam glass as an aggregate. Foam glass, a porous material made from recycled glass, offers excellent thermal and sound insulation properties, but its application in structural concrete remains unexplored [3-5]. This research aims to address this gap by investigating the feasibility of producing structural lightweight concrete using granular and crushed foam glass.

Foam glass is a lightweight, has a glass matrix containing millions of gas-filled cells [6-8]. It is fire-resistant, non-toxic, and can be tailored to have either open or closed porosity, depending on the application. Closed-pore foam glass is ideal for thermal insulation, while open-pore

foam glass is suitable for sound insulation [9]. The production of foam glass involves grinding waste glass into powder, adding a foaming agent, and heating the mixture to create a porous structure [10-12]. Despite its potential, the use of foam glass in structural concrete has been limited due to challenges in controlling its properties during production. A key knowledge gap is whether foam glass made from virgin glass differs significantly from that made from waste glass in terms of performance and durability. This research explores the differences and their implications for concrete applications.

The particle size of raw materials significantly influences the properties of foam glass, including cell size, density, compressive strength, and thermal conductivity. Smaller particle sizes generally lead to more uniform cell distribution and higher density, but excessively small particles can inhibit gas cell formation, reducing porosity [13]. Previous studies have used fullfactorial experimental designs to investigate these effects, which is very time, energy and material-intensive [4-6, 11, 13-28]. A knowledge gap exists in the application of statistical design of experiments (DOE), such as the Taguchi method, to optimize foam glass production. This research employs the Taguchi method to reduce the number of experiments while maintaining precision, offering a more efficient approach to optimizing foam glass properties. Foaming agents play a critical role in determining the properties of foam glass [17, 29-31]. Common foaming agents include SiC, calcium carbonate, and carbon black, which release gases like CO<sub>2</sub> during decomposition [4, 17, 32-35]. The choice of foaming agent affects the type of gas produced, the porosity, and the mechanical properties of the foam glass [36]. A novel approach in this research is the use of soot from cement factories as a foaming agent. Soot, a waste product rich in carbon black and calcium carbonate, has the potential to produce foam glass with uniformly distributed cells, low density, and high compressive strength. However, no previous studies have explored the use of cement factory soot for this purpose. This research investigates the combined use of soot and silicon carbide as foaming agents, along with alumina as a modifying additive, to enhance foam glass properties.

The production of foam glass typically involves pressing and molding glass powder, which is time-consuming and energy-intensive [39-37]. A novel approach in this research is the addition of water to the precursor powder to eliminate the dry pressing step. While previous studies have used water glass in foam glass production [27, 37, 40], this is the first study to investigate the effect of adding only water. This innovation could streamline the production process and reduce energy consumption.

The application of foam glass as an aggregate in concrete has been limited to non-structural applications, primarily due to the use of crushed foam glass, which increases open porosity and reduces workability [41-46]. Granulated foam glass, with its spherical shape and smooth

surface, offers a potential solution to these issues. A significant knowledge gap is whether granulated and crushed foam glass can be used to produce structural lightweight concrete with a compressive strength of at least 17 MP [47]. This research aims to address this gap by developing a mix design for structural lightweight concrete using granulated foam glass.

The production of granular foam glass in laboratory settings is challenging due to the complexities of controlling the foaming process. Previous studies have not provided a comprehensive method for producing foam glass granules with uniform cellular structure and minimal open porosity. This research introduces a novel laboratory method for producing granular foam glass, addressing this knowledge gap and enabling further exploration of its applications in concrete.

The alkali-silica reaction (ASR) is a major concern in concrete containing reactive silica, such as foam glass. ASR can lead to cracking and deterioration over time [48]. While previous studies have used long-term methods to assess ASR, this research proposes a novel approach using thermogravimetric analysis (TGA) to predict ASR reactivity without the need for concrete samples [49]. This method, developed in collaboration with Professor Marco Valenta's team at Sapienza University, represents a significant innovation in assessing the durability of foam glass concrete.

In conclusion, this research addresses several critical knowledge gaps in the field of lightweight concrete and foam glass, including the role of granulated and crushed foam glass in producing structural lightweight concrete, optimization of foam glass production, and assessment of ASR reactivity. By exploring these areas, this study aims to contribute to the development of sustainable and high-performance construction materials.

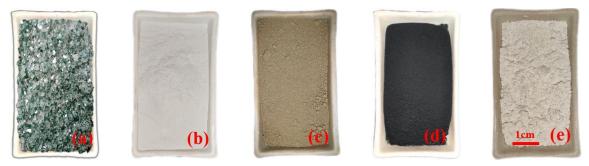
#### 3. Materials and methods

This section outlines the experimental methods and materials used to investigate the potential of foam glass granules as lightweight aggregates in structural concrete. The research focuses on optimizing foam glass production, characterizing its properties, and developing a concrete mix design using the ACI method. The methodology spans from raw material characterization to the fabrication of foam glass granules and their application in lightweight concrete.

#### 3.1. Raw materials and characterization

The primary materials used in this study include waste window glass, virgin glass powder, silicon carbide (SiC), cement factory soot, and alumina. These materials were characterized using various techniques to determine their physicochemical properties. Particle size distribution was analyzed using a laser particle size analyzer, while mineral phase composition was evaluated using X-ray diffraction (XRD). The chemical composition of the materials was determined using X-ray fluorescence (XRF), and morphological characteristics were examined

using scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS). Thermal behavior was studied using thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Heating microscopy was employed to investigate the sintering behavior of foam glass preforms.



**Figure 1.** foam glass raw material a) Waste glass particles, b) virgin glass powder, c) SiC, d) Soot, e) Al<sub>2</sub>O<sub>3</sub>, photos are taken by mobile phone camera (iPhone 13 pro max).

#### 3.2. Foam glass production and optimization

The production of foam glass involves mixing glass powder with foaming agents (SiC/Soot) and additives (alumina). The Taguchi design of experiments (DOE) method was employed to optimize the particle size, foaming agent content, sintering parameters, and molding type. The experiments were designed using orthogonal arrays (L8, L9, and L18) to minimize the number of trials while maximizing data output. Two fabrication methods were explored: the dry powder method, which involves pressing the powder mixture under high pressure, and the wet powder method, which uses water as a binder to eliminate the need for dry pressing. The optimal sintering temperature and holding time were determined using heating microscopy, and the resulting foam glass samples were tested for density, water absorption, open porosity, thermal conductivity, and compressive strength.

#### **3.2.1. Sintering parameters**

The effects of sintering temperature and holding time were investigated using an L18 orthogonal array. The sintering temperatures were derived from heating microscopy results, and holding times of 10, 30, and 50 min were tested.

#### 3.2.3. Fabrication methods

Two methods were used for foam glass production in this study are shown in Figure 2.

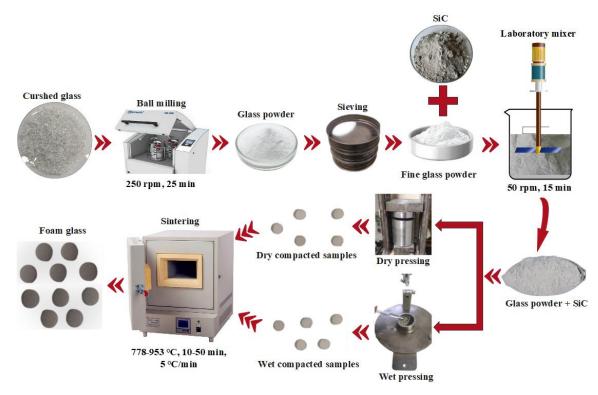


Figure 2. The procedure of dry powder molding for producing foam glass

And the influence of each technique was investigated according to the L8 Taguchi design of experiments.

#### 3.2.4. Using cement factory soot and alumina

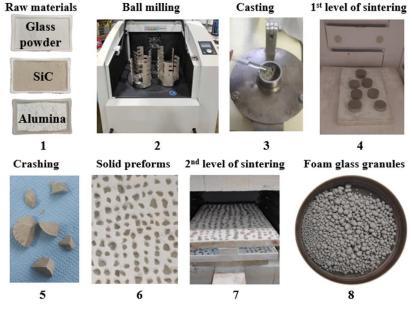
The effects of soot and alumina on foam glass properties were investigated using an L9 Taguchi orthogonal array. The samples were sintered at two temperatures (842°C and 890°C) with a holding time of 10 minutes.

#### 3.2.5. Selection of optimal foam glass

A multi-criteria decision-making (MCDM) approach, specifically the weighted sum model (WSM), was used to select the optimal foam glass composition based on density, thermal conductivity, water absorption, compressive strength, and expansion.

#### 3.3. Granular foam glass production

A novel laboratory method was developed to produce foam glass granules. This method involves a two-step sintering process: first, the glass powder is sintered at the glass transition temperature (Tg) to form a brittle preform, which is then crushed and refired at a lower temperature to produce granules with minimal open porosity (Figure 3). The granules were characterized for density, water absorption, and compressive strength. XRD and SEM analyses were conducted to examine the microstructure and crystalline phases of the granules.



**Figure 3.** The process of making foam glass granules

#### 3.5. Multi-criteria decision making for optimal foam glass

A multi-criteria decision-making (MCDM) approach was used to select the optimal foam glass composition and fabrication method. The WSM was applied, considering criteria such as density, thermal conductivity, water absorption, compressive strength, and expansion. The optimal foam glass was chosen based on its suitability for use in structural lightweight concrete.

#### 3.6. Alkali-silica reactivity (ASR) assessment of foam glass granules

A novel TGA-based method was developed to assess the alkali-silica reactivity of foam glass aggregates. This method involves preparing a paste of foam glass powder and calcium hydroxide, hydrating it for 1, 7, and 28 days, and analyzing the samples using TGA. The presence of a smooth TGA curve at 500°C indicates alkali reactivity, while a sharp peak suggests non-reactivity. This method provides a rapid and efficient alternative to conventional ASR testing. Figure 4 illustrates the described process.



**Figure 4.** The procedure of testing ASR using a TGA device

#### 4. Results and discussion

#### 4.1. Raw materials characterization results

The raw materials used in the production of foam glass for structural lightweight concrete were characterized to assess their physical, chemical, morphological, and thermal properties, with particular attention to their suitability for foam glass synthesis. The materials studied included virgin and waste glass powders, soot, alumina, and silicon carbide (SiC). Particle size analysis revealed that virgin glass powder had a mean particle size (D50) of 29.9 µm, which was significantly coarser than that of alumina (2.2 µm) and soot (4.7 µm); SiC particles were smaller than 1 µm and thus beyond the detection range of the analyzer. The smaller particle sizes of alumina and soot may enhance homogeneity and packing density in mixtures. X-ray diffraction (XRD) analysis showed that both virgin and waste glass powders were entirely amorphous, confirming their suitability for foam glass production. Soot contained calcite as the primary crystalline phase with minor dolomite, while alumina consisted of crystalline corundum (Al<sub>2</sub>O<sub>3</sub>), and SiC showed distinct crystalline silicon carbide peaks. X-ray fluorescence (XRF) results confirmed that virgin and waste glass powders were primarily composed of SiO<sub>2</sub> (72.67 wt% and 70.58 wt%, respectively), with minor constituents including Na<sub>2</sub>O and CaO. Soot was found to be mainly composed of CaO (49.35 wt%) with a high loss on ignition (38.61 wt%), indicating the thermal decomposition of calcium carbonate and its potential as a foaming agent. Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) analyses revealed that glass powders had irregular, angular shapes with elemental compositions dominated by silicon, sodium, and calcium. Soot particles were finer and more uniform, with calcium as the major element, while alumina showed fine and

consistent particles primarily composed of aluminum and oxygen. SiC particles were also very fine and exhibited high concentrations of silicon and carbon. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of soot showed a broad decomposition range, with weight losses attributed to water evaporation, volatile decomposition, and the breakdown of calcium carbonate into CaO and CO<sub>2</sub> between 474°C and 815°C. This thermal behavior confirms the role of soot as a potential foaming agent, although its wide decomposition range may lead to the formation of interconnected pores, influencing the final structure and properties of the foam glass. Overall, the raw materials demonstrated the necessary properties for effective application in foam glass production, with each material contributing uniquely to the process.

#### 4.2 Foam glass characterization results

#### 4.2.1 Investigation of samples' maximum height and area on heating microscopy images

Heating microscopy was employed to monitor the dimensional changes in foam glass samples during thermal treatment, specifically observing variations in height and area as temperature increased. It was found that the temperature corresponding to the maximum area expansion did not align with the temperature at which the greatest height increase occurred, indicating that the temperature of maximum expansion is more accurately determined using area-based measurements rather than height-based ones. To further investigate the influence of sintering parameters, the effects of temperature and holding time on the microstructure and properties of foam glass were examined. As sintering temperature increased, cell sizes grew larger and became more dispersed, while holding time had a minor influence on cell size but contributed to greater uniformity in cell distribution. Foam glass density initially decreased with rising temperature, reaching a minimum at around 876 °C, beyond which it began to increase due to pore collapse and partial melting of internal crystals. Similarly, compressive strength declined with higher temperatures, attributed to the formation of larger pores that weaken the structure. Open porosity and water absorption generally rose with temperature, although at very high temperatures (953 °C), both properties dropped due to the collapse of pore structures. Thermal conductivity followed a similar trend, decreasing as sintering temperature increased up to 876 °C and then rising again as a result of reduced porosity and increased solid-phase connectivity from cell wall collapse. These findings highlight the critical role of sintering parameters in tailoring the final properties of foam glass.

#### 4.2.2. Effect of molding type on foam glass samples

The influence of molding methods, specifically dry powder and wet powder techniques, on the properties of foam glass was systematically examined. Wet-powder samples were found to

develop a greater number of well-defined crystals in their microstructure compared to those produced by the dry-powder method, indicating enhanced crystallization under wet processing conditions. In terms of density, both molding methods showed a similar trend: density decreased with rising sintering temperature up to 876 °C, after which it increased due to cell collapse and partial melting. However, wet-powder samples demonstrated higher compressive strength than dry-powder samples, likely due to improved homogeneity and interparticle bonding. Open porosity and water absorption increased with higher sintering temperatures, reflecting expanded pore structures, but both properties declined at extremely high temperatures (such as 953 °C) as a result of pore collapse. Thermal conductivity exhibited an inverse relationship with porosity; lower porosity at higher temperatures led to increased thermal conductivity, again attributed to the collapse of insulating gas-filled cells and the resulting enhancement of heat transfer through the solid matrix. These findings highlight the significant role of molding method in shaping the structural and thermal performance of foam glass.

The results indicate that sintering temperature had the most significant influence on foam glass properties (69%–92%), followed by the molding method (3%–18%) and holding time (4%–13%).

#### 4.2.4. Effect of using virgin glass and alternate additives (alumina, cement factory soot)

The influence of alumina and cement factory soot, on the properties of foam glass was thoroughly investigated. The incorporation of alumina into the glass matrix enhanced the viscosity of the softened glass during sintering, which restricted cell growth and resulted in smaller, more uniform cells. This refinement of the microstructure contributed to a notable increase in compressive strength. In contrast, soot served as a foaming agent due to its thermal decomposition; however, because it decomposed over a broad temperature range, it generated interconnected cells that could compromise the mechanical integrity of the foam. In terms of density, samples containing higher amounts of alumina exhibited increased density, whereas greater quantities of soot led to lower density due to excessive foaming. Water absorption was inversely related to alumina content, as increased alumina reduced open porosity and thereby limited moisture uptake. Similarly, thermal conductivity rose with higher alumina content, a consequence of decreased porosity and enhanced solid-phase connectivity. Compressive strength trends mirrored those of density and porosity: strength increased with the addition of alumina but decreased as the amount of foaming agent rose. These results underscore the

critical role of additive selection and proportion in tailoring the physical, thermal, and mechanical properties of foam glass.

#### 4.2.5. Results of novel fabrication method for making foam glass granules

A novel method for producing foam glass granules was developed, involving sintering at the glass transition temperature (Tg) and re-sintering at a temperature 20°C below the maximum expansion temperature. The results, shown in Figure 3, indicate that the method successfully produced foam glass granules with low open porosity and high compressive strength. The optimal compositions for use in lightweight concrete were determined to be 2 wt% alumina, 2 wt% SiC, and 96 wt% virgin glass (sample 1) and 2 wt% alumina, 2 wt% SiC, and 96 wt% waste glass.

#### 4.3. Performance of granulated foam glass as concrete aggregate

This section presents the results of using granulated foam glass as a replacement for natural aggregates in lightweight concrete. The focus is on the mix design, compressive strength, failure modes, alkali-silica reaction (ASR), and the application of a novel TGA-based method for assessing ASR reactivity.

#### 4.3.1. Lightweight concrete mix plan

The ACI method was used to design the concrete mix, with varying percentages of natural coarse aggregates replaced by foam glass granules (20%, 40%, and 60%). The mix proportions are detailed in Table 1. The cross-sections of the concrete samples demonstrate excellent adhesion between the cement paste and both natural and foam glass aggregates.

Table 1. Specimens of concrete mix plans in the sample with dimensions of 5cm×5cm ×5cm

|                       |           | Sam        | ple type   |            |  |  |  |  |
|-----------------------|-----------|------------|------------|------------|--|--|--|--|
| Ingredients           | Reference | 20% Coarse | 40% Coarse | 60% Coarse |  |  |  |  |
| Water (gr)            | 25.25     | 25.25      | 25.25      | 25.25      |  |  |  |  |
| Cement (gr)           | 33.50     | 33.50      | 33.50      | 33.50      |  |  |  |  |
| Coarse aggregate (gr) | 129.87    | 104.00     | 77.92      | 51.95      |  |  |  |  |
| Fine aggregate (gr)   | 47.75     | 47.75      | 47.75      | 47.75      |  |  |  |  |
| Coarse FGA (gr)       | 0.00      | 2.81       | 5.43       | 8.15       |  |  |  |  |

#### Density and compressive strength of cement matrix composite

The density and compressive strength of the concrete samples were measured after 1 day and 28 days of curing. The results are summarized in Table 2:

**Table 2.** The results of fresh concrete density, air-dried cement matrix composite density, and compressive strength of the samples

|             | Density (kg/m³) |        |       |      | Compressive strength (MPa) |        |       |      |
|-------------|-----------------|--------|-------|------|----------------------------|--------|-------|------|
| Sample type | Rep. 1          | Rep. 2 | Rep.3 | Avg. | Rep. 1                     | Rep. 2 | Rep.3 | Avg. |
| Reference   | 2160            | 2160   | 2170  | 2160 | 28                         | 31     | 29    | 29   |
| 20% coarse  | 1850            | 1840   | 1910  | 1860 | 20                         | 20     | 21    | 20   |
| 40% coarse  | 1640            | 1700   | 1660  | 1660 | 14                         | 19     | 13    | 15   |
| 60% coarse  | 1550            | 1520   | 1540  | 1540 | 9                          | 10     | 8     | 9    |

All lightweight concrete samples had densities below 2000 kg/m³, meeting the requirements for structural lightweight concrete. Only the 20% foam glass replacement sample met the ISO 22965-1:2007 structural strength criterion of 17 MPa. The 40% and 60% replacement samples had lower compressive strengths, failing to meet the structural requirements.

#### 4.3.2. Alkali-silica reaction (ASR) of concrete

The alkali-silica reactivity (ASR) of the concrete samples was assessed in accordance with ASTM C1260. The results revealed that all samples exhibited expansions below 0.22%, classifying them as moderately reactive based on the ASTM C1778 criteria. A comparison between granulated and crushed foam glass aggregates demonstrated that concrete containing granulated foam glass exhibited lower expansion, suggesting that granulated foam glass possesses lower reactivity than its crushed counterpart. Furthermore, SEM analysis showed no evidence of silica gel formation at the interface between the foam glass and the cement matrix, indicating that the foam glass aggregate itself is inert. Therefore, the observed expansion is likely attributable to ASR reactions involving other reactive components within the concrete matrix.

#### 4.3.3 Compressive strength of cement matrix composite samples

The compressive strength of the concrete samples was evaluated. Concrete incorporating granulated foam glass demonstrated higher compressive strength compared to that containing crushed foam glass, highlighting the structural advantage of granulated particles. Additionally, although waste-derived foam glass granules exhibited inherently higher compressive strength as individual particles, the concrete produced with virgin foam glass granules achieved superior overall strength. This discrepancy is attributed to the formation of ettringite crystals within the waste foam glass granules during hydration, which induced internal cracking and consequently reduced the mechanical performance of the concrete.

#### 4.3.4 TGA analysis for ASR assessment

A novel thermogravimetric analysis (TGA)-based approach was employed to evaluate the alkali-silica reaction (ASR) reactivity of foam glass aggregates. The findings demonstrate that all foam glass samples exhibited pozzolanic activity, as evidenced by their ability to fix calcium hydroxide [Ca(OH)<sub>2</sub>], which increased with curing time. This behavior suggests that foam glass can effectively reduce the concentration of free lime in the cement matrix, thereby mitigating ASR-induced expansion and

cracking. Regarding the influence of sintering conditions, sintering cycles had a negligible impact on the pozzolanic reactivity of foam glass derived from virgin glass. In contrast, foam glass produced from waste glass exhibited enhanced pozzolanic reactivity after a second sintering cycle, likely due to the chemical inhomogeneity of the waste glass composition.

#### 5. Conclusion

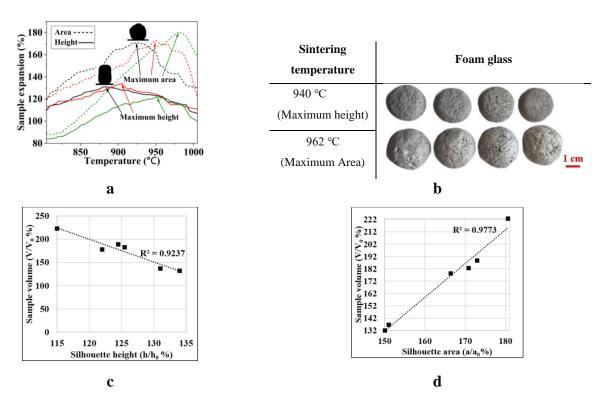
This study yielded several significant findings on the development and application of foam glass aggregates for structural lightweight concrete:

- Heating microscopy analysis showed that maximum surface area expansion occurred 18–60 °C above the temperature of maximum height change, and can estimate the maximum expansion better than maximum height.
- Sintering parameters revealed that temperature was the dominant factor, influencing 79% of foam properties, while holding time contributed 21%.
- Adding 20 wt% water to the foam glass precursor powder could eliminate the need for cold pressing (>80 MPa), with changes of 3% in porosity, 16% in density, and 18% in compressive strength.
- Soot (up to 3%) acted as an effective waste foaming agent, enhancing expansion and compressive strength.
- An optimized foam glass formulation, virgin glass sintered at 30 °C below the peak of heating microscope temperature (maximum height) with 8% alumina, 1% soot, and 2% SiC (wet powder method), was identified using multi-criteria analysis.
- A new lab-scale granulation method was developed, producing nearly spherical foam glass granules with varied sizes.
- Structural lightweight concrete was successfully produced by replacing 20 vol% of natural aggregates with foam glass granules, achieving <2000 kg/m³ density and >17 MPa strength, fulfilling ISO 22965-1:2007 structural requirements.
- Concrete with foam glass granules (both virgin and waste) outperformed those made with crushed foam glass in compressive strength.
- Despite higher granule strength in waste glass, ettringite-induced cracking led to stronger concrete with virgin glass granules.
- ASR tests showed all mixes were moderately reactive, with lower expansion in granulated foam glass than crushed, and suitable for environments with ASR risk ratings of 1–2 out of 6.
- A novel TGA-based method confirmed that foam glass aggregates exhibit good pozzolanic reactivity while remaining non-reactive toward ASR, validating their compatibility with cement matrices.

#### 6. Claims and new scientific results

## 1. Comparative evaluation of foam glass expansion based on maximum silhouette height and maximum projected area temperatures extracted from heating microscopy images

I conducted a comparative evaluation of foam glass characteristics at two distinct temperatures derived from heating microscopy images: one corresponding to the maximum silhouette height and the other to the maximum projected 2D area. Foam glass samples were synthesized using waste window glass combined with 1–3 wt% SiC as the foaming agent. My experimental results demonstrated that sintering at the temperature associated with maximum projected area, typically 18–60 °C higher than that of maximum height, consistently resulted in significantly greater volumetric expansion, ranging from 44% to 57%. In addition, regression analyses revealed a stronger correlation between the projected area ratio (a/ao) and volumetric expansion ( $R^2 = 0.9773$ ), compared to the height ratio (h/ho) ( $R^2 = 0.9237$ ). These findings highlight the superior predictive capability of the area-based method in identifying the true temperature of maximum expansion. This comparative investigation offers a more accurate and practical approach for determining optimal sintering temperatures in foam glass production through heating microscopy analysis.

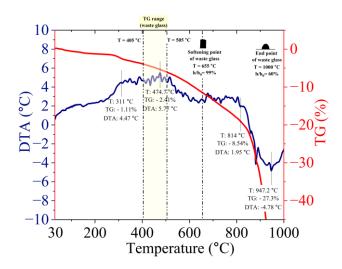


**Figure C1.** a) The difference in area and height changes graphs for samples made from waste glass powder and, 1 wt% (green lines), 2 wt% (red lines) and 3 wt% (black lines) of SiC. b) Comparison of the same foam glass (1 wt% SiC + waste window glass powder) samplese, which are sintered at maximum height and maximum area temperatures. c) Correlation between height ratio ( $h/h_0$ ) and volume ratio ( $h/h_0$ ) of foam glass samples. d) Correlation between area ratio ( $h/h_0$ ) and volume ratio ( $h/h_0$ ) of foam glass samples.

## 2. Application of waste soot originating from a cement factory as a foaming agent in foam glass production

**2.1.** I demonstrated that cement factory soot, a waste material rich in carbon and calcium carbonate (CaCO<sub>3</sub>), can be employed as a dual-phase gas-releasing agent in foam glass production. Carbon undergoes oxidation at 475 °C (as confirmed by Thermogravimetry analysis in Figure C.2.1), releasing CO<sub>2</sub> and initiating primary foaming. Subsequently, above 815 °C, CaCO<sub>3</sub> starts to decompose and releases additional CO<sub>2</sub>, contributing to a secondary foaming. However, this approach requires the glass matrix to be sufficiently softened at the first gas release phase; based on heating microscopy results, the glass transition temperature is in the range of (405 to 505 °C), and the softening point of the glass is about 655 °C. Therefore, the first-stage CO<sub>2</sub> evolution from carbon leads to partial pre-foaming and bubble nucleation, but complete swelling requires temperatures higher than 814 °C.<sup>1</sup>

With just 2 wt% soot, foam glass with a low bulk density of 241 kg/m³ was achieved at 840°C. In comparison, a previous study using 4 wt% CaCO₃ at 820°C resulted in a higher density of 301 kg/m³, approximately 16% more [50]. Importantly, this method promotes waste valorization and aligns with circular economy goals. Therefore, the use of cement factory soot as a foaming agent can be considered highly practical and industrially applicable.

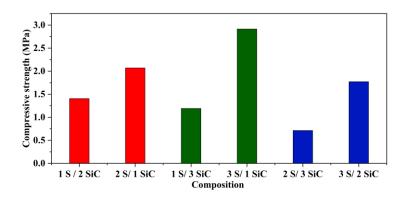


**Figure C2.1.** Thermogravimetric analysis (TGA) curve of soot alongside the critical temperature points of waste glass. The glass transition temperature of the waste glass is observed in the range of 405°C to 505°C, and its softening point occurs at approximately 655°C. The oxidation temperature of carbon and the decomposition temperature of calcium carbonate in the soot are also clearly indicated on the curve.

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<sup>&</sup>lt;sup>1</sup> The results discussed in this claim have been published in the Q1 journal *Ceramics* International and are accessible at <a href="https://doi.org/10.1016/j.ceramint.2024.10.152">https://doi.org/10.1016/j.ceramint.2024.10.152</a>.

**2.2.** My experimental results demonstrate that the use of cement factory soot as a foaming agent, particularly in combination with silicon carbide (SiC), enhances the compressive strength of foam glass. In our published study [36], I investigated the effects of varying soot and SiC contents (1, 2, and 3 wt%) and found that this hybrid foaming strategy leads to significantly improved mechanical performance. As shown in previous research (summarized in Figure C.2.2, based on the literature), calcium carbonate has traditionally been used as a foaming agent for recycled window glass, resulting in compressive strengths ranging from 0.52 to 6.5 MPa depending on the heating regime and foaming agent dosage [51-53]. In comparison, foam glass produced using cement factory soot achieved compressive strengths between 0.86 and 9.2 MPa [36], representing a notable enhancement.



**Figure C2.2.** Compressive strength variations of foam glass produced from waste glass using a combined foaming agent of soot and SiC. The symbol 'S' denotes soot, and the numbers following it indicate the corresponding weight percentage used in the foam composition.

#### 3. Introduction of a simplified laboratory method for making foam glass granules

My research introduces a novel, two-step sintering method for producing foam glass granules, which eliminates the need for pelletizers and rotary furnaces (Figure C3). This method allows for the production of foam glass granules in a wide range of sizes with a uniform pore distribution. The use of the two-step sintering process, which is a significant departure from conventional methods, enables the creation of foam glass granules with a structure resembling that of typical commercial foam glass, without the need for specialized equipment. The outer shell of aggregates in this production method is continuous, with no significant cracks or voids, resulting in improved mechanical strength and a uniform structure.

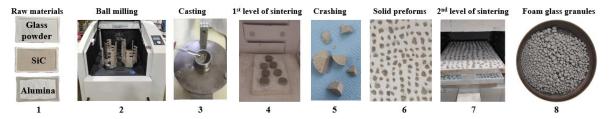
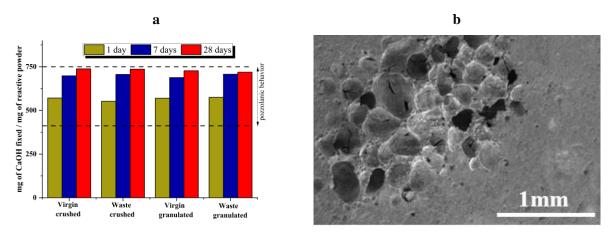


Figure C3. The novel technique for fabricating foam glass granules

## 4. Application of Thermo-Gravimetric Analysis (TGA) as a predictive tool for indirectly assessing Alkali-Silica Reaction (ASR) by foam glass in cementitious environment<sup>2</sup>

**4.1.** A new experimental approach was introduced in this research to assess the reactivity of foam glass toward calcium hydroxide Ca(OH)<sub>2</sub> as an indirect indication of ASR mitigation potential. In this method, foam glass powder was mixed with reagent-grade Ca(OH)<sub>2</sub> in equal proportions by mass and hydrated under controlled conditions. After specified curing durations (1, 7, and 28 days), the residual unreacted Ca(OH)<sub>2</sub> was quantified using thermogravimetric analysis. The absence or reduction of the characteristic decomposition peak of Ca(OH)<sub>2</sub> at approximately 500 °C is interpreted as an indicator of pozzolanic activity and potential mitigation of ASR. The evaluation criterion is based on the amount of fixed Ca(OH)<sub>2</sub>, expressed in milligrams per gram of reactive material. These results were benchmarked using values established in previous literature [54], where glass materials fixing more than 436 mg Ca(OH)<sub>2</sub>/g are considered pozzolanic. The procedural steps of this novel TGA-based method are illustrated in Figure C4.1.

**4.2.** Experimental results showed that foam glass powders exhibit significant pozzolanic reactivity by absorbing free Ca(OH)<sub>2</sub> from the cement paste (Figure C4.2a) and contributing to the formation of calcium-silicate-hydrate (C–S–H) gel. This reduces the availability of free Ca<sup>2+</sup> and OH<sup>-</sup> ions in the pore solution, thereby lowering alkalinity and decreasing the risk of ASR (Figure C4.2b).



**Figure C4.2.** a) Calcium hydroxide fixation rate, indicating the pozzolanic reactivity of foam glass particles, as determined by TGA analysis, b) SEM image showing the non-reactive interface between foam glass aggregate and cement paste, with no evidence of ASR gel formation after exposure to ASR testing conditions.

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<sup>&</sup>lt;sup>2</sup> The results discussed in this claim have been published in the D1 journal (Journal of Building Engineering). DOI: 10.1016/j.jobe.2025.112270

#### **Publications**

- Properties of foam glass produced with the use of soot from a cement factory as a foaming agent: A study based on Taguchi design of experiments, Masoud Osfouri, Jamal-Eldin FM Ibrahim, Andrea Simon, 2024/12/15, Ceramics International (Q1 journal), Volume 50: Issue 24, <a href="https://doi.org/10.1016/j.ceramint.2024.10.152">https://doi.org/10.1016/j.ceramint.2024.10.152</a>.
- ❖ Sustainable Structural Lightweight Concrete Containing Foam Glass Aggregates, Masoud Osfouri, Jamal-Eldin FM Ibrahim, Matteo Sambucci, Marco Valente, Jacopo Tirillò, Simon Andrea, 2025/3/5, Journal of Building Engineering (**D1 journal**), Volume 104, https://doi.org/10.1016/j.jobe.2025.112270.
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